International Journal of Thermophysics

Development of Thermophysical Handy Tester for Non-destructive Evaluation of Engineering Solid Materials[1]

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1 Paper presented at the Fifteenth Symposium on Thermophysical Properties, June 22-27, 2003, Boulder, Colorado, U.S.A.

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ABSTRACT

Techniques of quality inspection and non-destructive diagnostic of engineering materials have been developed as the procedures determining some kinds of physical properties of materials. The diagnostic techniques are usually based on detecting a very delicate signal concerned with changeable properties, and therefore many experiences on the procedure are required in order to detect such signal. In si-tu measurement for thermophysical properties of engineering parts is also important for judgment of intrinsic soundness of the parts or for distinction between similar materials. The purpose of this study is the development of a thermophysical properties handy tester which is able to make simultaneous measurement of thermal conductivity and thermal effusivity, and applicable to the non-destructive diagnostic of many engineering materials. The tester consists of a portable data-acquisition unit, a probe holder equipped with a thermal probe, and a note computer. The measurement can be made merely by the probe point-contacting on a testing body for the period of 10 seconds. The thermal probe is constructed with a thin thermocouple so as to satisfy some conditions for the measurement principle. Materials which can be measured are polymeric resins, glasses, ceramics, alloys, pure metals and so on.

KEY WORDS: comparative method; in-situ measurement; point-contact; solids; thermal conductivity; thermal effusivity.

1. INTRODUCTION

Utility of the thermophysical properties information for the technique of quality inspection or distinction between similar materials has been noticed recently. Although a strict measurement method is important to know accurate thermophysical properties, an easy measurement method is also necessary for in si-tu measurement, such as non-destructive quality inspection of industrial materials. Most existing measurement methods of thermophysical properties need some kinds of test pieces and thereby it is impossible to apply them to in si-tu measurement.

Measurements of the thermal conductivities of dielectric thin films using the thermal comparator method [1,2] have been reported. The comparator method has a possibility for in si-tu measurement because of a point contact method. In this method, however, there are some problems that the contact pressure must be constantly kept, and applicable condition is also severer for surface quality, such as hardness.

A measuring method of three thermophysical parameters of solids by a thermal probe with instantaneous point contact [3,4] has been recently developed. This method will be widely used in the industry field, because ready measurement is possible. Therefore, the purpose of this study is practically to construct a thermophysical properties handy tester based on this method in order to carry out in si-tu measurement. The newly developed tester has several advantages over the thermal comparator, not the least of which is an instantaneous measurement method of three thermophysical parameters (thermal conductivity, thermal diffusivity, and specific heat capacity) of solids. The tester consists of a portable data-acquisition unit with dc-power supply, a probe holder equipped with a thermal probe, and a note computer. The measurement can be made merely by the probe point-contacting on a body for the period of 10 seconds. The probe of this tester is composed of a thin thermocouple of the type-E so as to satisfy measuring conditions. For this tester, special test pieces are not necessary and testing bodies need not always have a plane surface on account of the point-contact measurement. Measurement accuracy depends upon that of the test measurement for determining thermal constants of the probe, since this is a kind of comparative measurement. Materials which can be measured are polymeric resins, glasses, ceramics, alloys, pure metals and so on. Typical applications are presented in this paper, such as the evaluation of an effective thermal conductivity of an alumina-FRP and the inspection of chemical composition heterogeneity of an inter-metallic compound alloy, NiAl-slab.

2. PRINCIPLE

Engineering solid materials are usually isotropic or orthotropic. Heat conduction equation for orthotropic medium in the rectangular coordinate system is given by

$$(\rho c)_e \frac{\partial T}{\partial t} = \lambda_x \frac{\partial^2 T}{\partial x^2} + \lambda_y \frac{\partial^2 T}{\partial y^2} + \lambda_z \frac{\partial^2 T}{\partial z^2} \tag{1}$$

New independent variables X, Y, Z are defined as

$$X = \left(\frac{\lambda_e}{\lambda_x}\right)^{\frac{1}{2}} \cdot x\ ,\quad Y = \left(\frac{\lambda_e}{\lambda_y}\right)^{\frac{1}{2}} \cdot y\ ,\quad Z = \left(\frac{\lambda_e}{\lambda_z}\right)^{\frac{1}{2}} \cdot z \tag{2}$$

where

$$\lambda_e = \left(\lambda_x \cdot \lambda_y \cdot \lambda_z\right)^{\frac{1}{3}}. \tag{3}$$

Moreover, in the case of the heat conduction by a point heat-source on a semi-infinite body, the spherical coordinate system which is set as $r = \left(X^2 + Y^2 + Z^2\right)^{1/2}$ is convenient. Therefore, equation (1) takes the form

$$\frac{\partial T}{\partial t} = a_e\left(\frac{\partial^2 T}{\partial r^2} + \frac{2}{r}\frac{\partial T}{\partial r}\right) \tag{4}$$

where the effective thermal diffusivity is defined as

$$a_e = \lambda_e /(\rho c)_e. \tag{5}$$

Now, suppose that a testing body is one of the parts in an industrial product and brought into point-contact with a tip of a thermal probe. Here, the tip is a junction bead of a thermocouple entered in the probe and maintained at a temperature higher than the room one before contacting. Then, we assume that heat flow occurs through a small contacting area of which shape is a hemi-sphere of the radius r_0, and the heat conducted into the testing body is expressed as

$$Q = 2\pi r_0^{\ 2}\left[-\lambda_e\left(\frac{\partial T}{\partial r}\right)\right]_{r=r_0} \tag{6}$$

The contacting surface of the thermal probe can be also assumed as the small hemi-sphere of the radius r_0, which is smaller than the tip of the probe. The transient temperature of the junction bead observed at the sensing point r_1 which is quite close to the contacting surface is said to be the temperature response of the thermal probe just after contacting. From the view point of a small area associated with such a temperature change, we obtain the analytical solution of the temperature response on the assumption that the tip of the probe is a semi-infinite body.

Heat conduction equations in both sides of the probe and the testing body are

$$\frac{\partial T_p}{\partial t} = a_p\left(\frac{\partial^2 T_p}{\partial r^2} + \frac{2}{r}\frac{\partial T_p}{\partial r}\right) \tag{7}$$

$$\frac{\partial T_s}{\partial t} = a_s\left(\frac{\partial^2 T_s}{\partial r^2} + \frac{2}{r}\frac{\partial T_s}{\partial r}\right) \tag{8}$$

where the thermal diffusivity of the probe is defined as $a_p = \lambda_p /(\rho c)_p$ and that of a testing body is $a_s = \lambda_s /(\rho c)_s$ instead of equation (5).

Conditions of continuity both temperature and heat flow on the contacting surface are

$$T_p(r_0, t) = T_s(r_0, t) \tag{9}$$

$$\lambda_p \left.\frac{\partial T}{\partial r}\right|_{r=r0} = -\lambda_s \left.\frac{\partial T}{\partial r}\right|_{r=r0} \tag{10}$$

Initial and boundary conditions are set as

$$T_p(r,0) = T_{p0}\,,\quad T_s(r,0) = T_{s0} \tag{11}$$

$$T_p(\infty,t) = T_{p0}\,,\quad T_s(\infty,t) = T_{s0} \tag{12}$$

where T_{p0} and T_{s0} are initial temperatures and uniform in each body, respectively.

Dimensionless temperature in the probe is defined as $T_p^* = (T_p - T_{p0})/(T_{s0} - T_{p0})$. By solving equations (7) to (12), the analytical temperature response at $r = r_1$ is obtained as follows:

$$T_p^* = \frac{\beta}{\eta(\beta+1)}\operatorname{erfc}\left(\frac{C}{\sqrt{t}}\right) + \frac{\zeta-\beta}{\eta(\zeta+1)(\beta+1)}\exp\left(X^2 - \frac{C^2}{t}\right)\operatorname{erfc}(X) \tag{13}$$

where

$$\beta = \frac{\lambda_s}{\lambda_p}\,,\quad \zeta = \frac{\xi_s}{\xi_p}\,,\quad \eta = \frac{r_1}{r_0}\,,\quad C = \frac{r_1 - r_0}{2\sqrt{a_p}}\,,\quad X = \frac{C}{\sqrt{t}} + \frac{(\beta+1)(\eta-1)}{2C(\zeta+1)}\sqrt{t} \tag{14}$$

In the period satisfying that $C/\sqrt{t} << 1$, equation (13) can be expressed as

$$T_p^* \cong \frac{\beta}{\eta(\beta+1)} - \frac{b}{(\beta+1)}\left\{\beta - \frac{\zeta-\beta}{\eta(\eta-1)(\beta+1)}\right\}\frac{1}{\sqrt{t}} \tag{15}$$

where $b = 2C/\sqrt{\pi}$.

This is implied that temperature response T_p^* is approximated to a linear function of $1/\sqrt{t}$ in the later period of it. Thus, the following relation can be applied to measured temperature responses in the similar period. That is,

$$T_p^* = A - B\frac{1}{\sqrt{t}} \tag{16}$$

If the parameters A and B in equation (16) can be found by least squares matching and the values of both b and η can be known, the thermal conductivity ratio β and the thermal effusivity ratio ζ can be calculated from equations (15) and (16) as follows:

$$\beta = \frac{A\eta}{1 - A\eta} \tag{17}$$

$$\zeta = (\beta+1)^2\left(A - \frac{B}{b}\right)\eta(\eta-1) + \beta \tag{18}$$

The procedure of determining the values of both b and η has been discussed in the previous paper [3]. This procedure takes a lot of calculating time. Therefore, using a fixed b , we can decide the value of η by curve-fitting the analytical temperature response, equation (13), with the measured one. The reason why b can be fixed is that the distance between the thermal sensing point of the probe and its contacting point with a testing body is constant for the case of using the same probe.

3. MEASUREMENT SYSTEM

Figure 1 schematically shows the newly developed thermophysical handy tester. The tester consists of a portable data-acquisition unit, a probe holder equipped with a thermal probe, and a note computer. Figure 2 shows the thermal probe within the probe holder.

Overall length of the holder is about 120mm. The probe holder can be manipulated in the one-

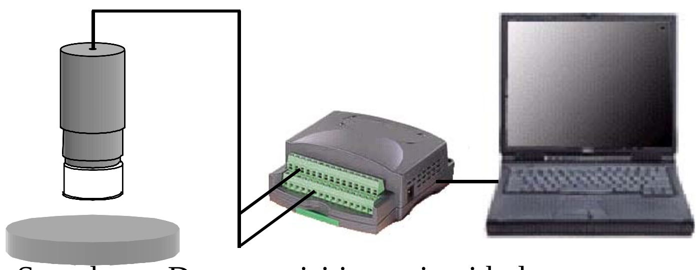

Fig. 1. Measuring system of the thermophysical handy tester

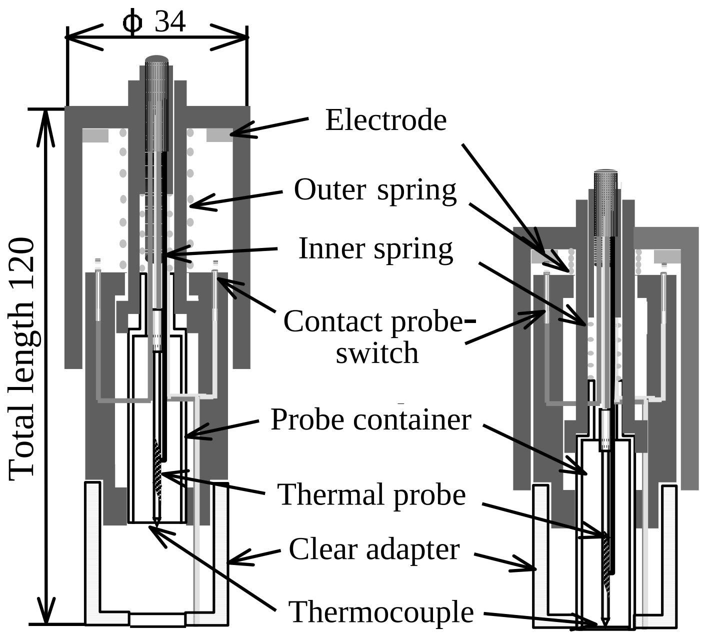

Fig. 2. Cross-sectional view of the probe holder

hand. The thermal probe is constructed using a thermocouple of the type-E and a wire of 0.1mm-diameter sheathed in a alumina tube of 0.8mm-diameter. The probe is kept to a temperature about 20℃ higher than a room temperature by a small heater installed in it. The measurement can be made merely by the probe point-contacting on a testing body for the period of 10 seconds. Then, the tip of the probe is extruded by depressing the holder from the opposite side. For the reduction in thermal resistance on the contacting surface, the contact pressure is made by an inner spring to be about 10 MPa. The temperature response is measured in the sampling frequency of 0.1 seconds. In order to start measuring just at contacting, the trigger system is used by the aid of a contact probe-switch built in the holder. The temperature responses are measured by a portable data-acquisition unit. And then, the data is converted into a CSV file and processed using the macro-function of EXEL on a note computer. As a result, the temperature response curve is drawn on the display with the result of the thermophysical properties calculated from the response.

4. MEASURED TEMPERATURE RESPONSES

For example, some temperature responses are plotted in figure 3. The thermophysical properties of these materials are known. It is shown that the response curves for many kinds of materials are clearly distinguishable.

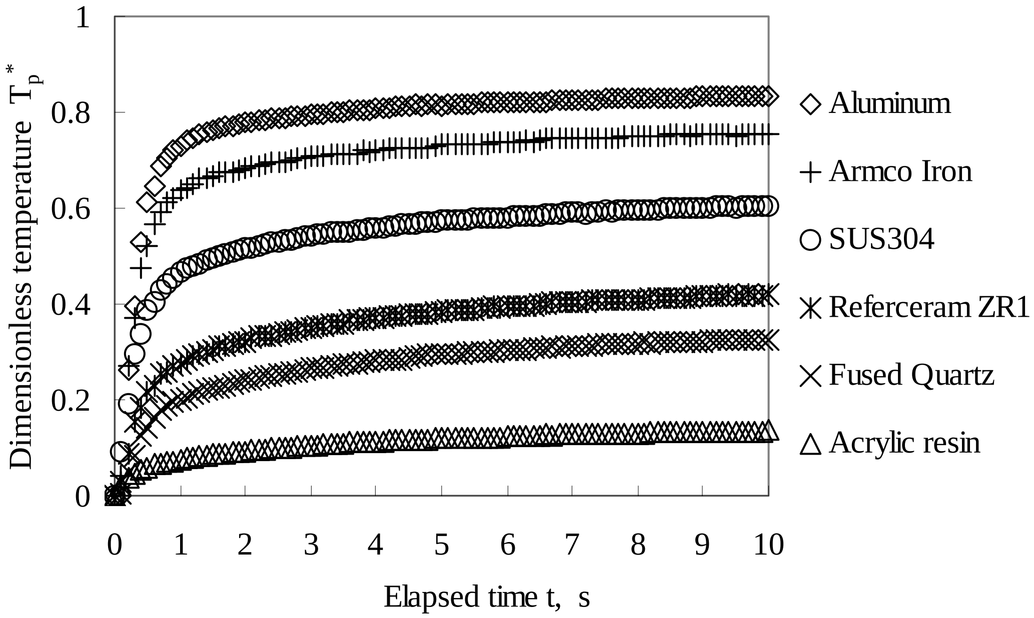

Fig. 3. Measured temperature responses for various materials

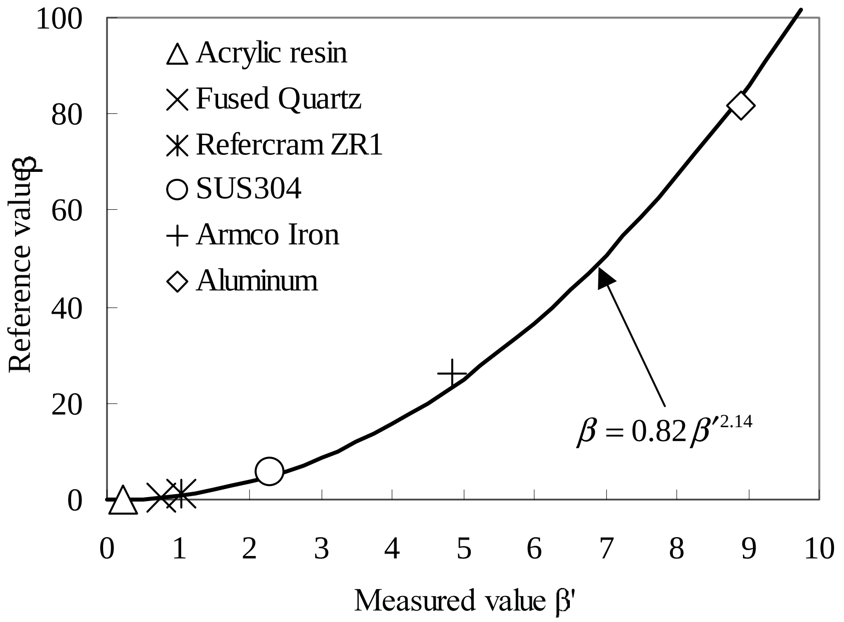

Fig. 4. Correlation of β between reference value and measured one

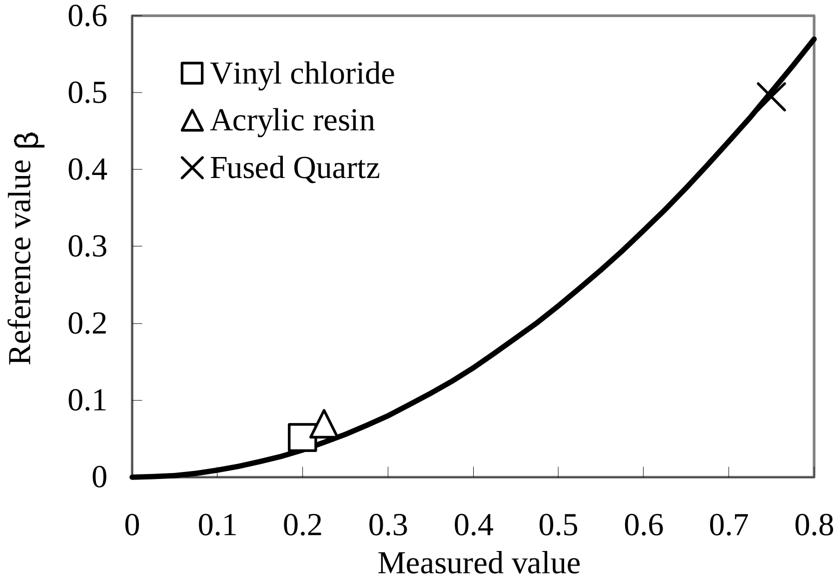

Fig. 5. Correlation of β between reference value and measured one in the case of the materials with a low thermal conductivity

These response curves are characterized according to four parameters, such as β, ζ, η, and b. This measurement method evaluates the bulk properties in the vicinity of the point-contacting surface to the depth of several millimeters. The value of η is decided by the size of the contact area in every measurement. On the other hand, the distance between the contacting point and the thermal sensing point of the probe does not vary, if the tip of the probe should not be transformed. Therefore, the value of *b* can be constant as long as the same probe is used. The radius of the contact area of the probe may be less than 0.2mm, and then, η is less than 1.1. In the result of analyzing these response curves, the value of *b* was determined as 0.0252[$s^{0.5}$] for this probe. Then, C = 0.0224 [$s^{0.5}$]. All of the response curves after the period of 5 seconds were ascertained to become rectilinear for $1/\sqrt{t}$.

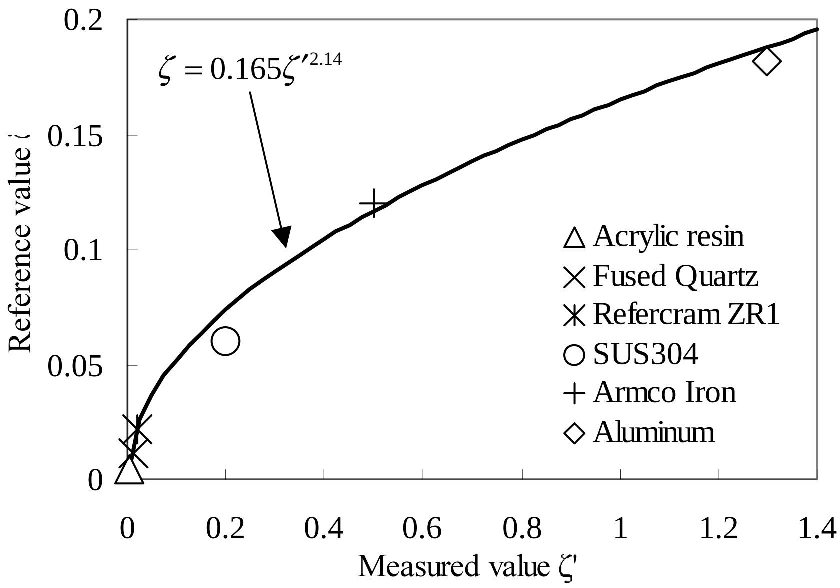

Fig. 6. Correlation of ζ between reference value and measured one

Figure 4 shows the correlation of β between measured value and reference one. The thermal conductivity of the probe was estimated from the measurement of austenitic stainless-steel, SUS304. In the case of the materials with a low thermal conductivity, the response is very reproducible and easy to analyze as shown in figure 5, so this method is useful to distinct polymeric resins.

Similar comparison for ζ is shown in Figure 6. The relation between measured value and reference value, that is the correction equation obtained by least-square method, is presented in each figure. A measured response curve has a systematic error due to sensitivity of a data-acquisition unit or unsatisfied conditions in the measurement principle, and thus it is necessary to correct the measured values of both β and ζ. If both β and ζ are obtained, the thermophysical properties, $\lambda_{s,}$ ξ_s of a testing body is known using those of the probe, i.e. λ_p=9.34[W・m^{-1}・K^{-1}], ξ_p=29.1[kJ・m^{-2}・s$^{-1/2}$・K^{-1}].

5. APPLICATIONS

Composite materials such as fiber reinforced plastics and inter-metallic compound alloys have been used in industrial products. The former is artificially made so as to be anisotropic materials. On the other hand, the latter is required to be homogeneous. For the quality inspection of such materials, the thermophysical handy tester has been applied.

Figure 7 shows the shape and coordinate system of sample of an alumina-fiber reinforced plastic. Diameter of the fiber is about 10μm and the content rate of the fiber is 70 vol.%. The sample is made like a die of 10mm-cube in superimposing the board of 1mm-thickness. It is obvious from the simulation shown in figure 8 that the configuration of temperature profile in the sample varies according to the direction of the fiber. Then, it is important that the diameter of a fiber is enough smaller than that of the contact area of the probe. It is obvious at a glance from figure 9 that the temperature response measured in the direction of z-axis is bigger than the other and the response curves in the directions of both x and y-axes agree with each other. It is also ascertained from this result that the effective thermal conductivity λ_e evaluated in the direction of x-axis is equal to that of y-axis because $\lambda_x = \lambda_y$ in equation (3). The effective thermal conductivity λ_e was obtained as 1.2 W/(m・K).

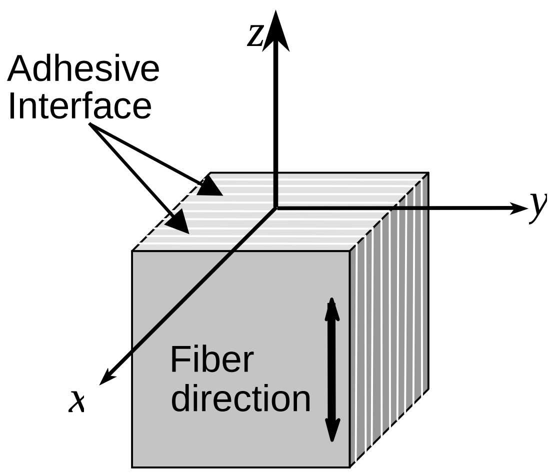

Fig. 7. Testing body of alumina-FRP and coordinate system

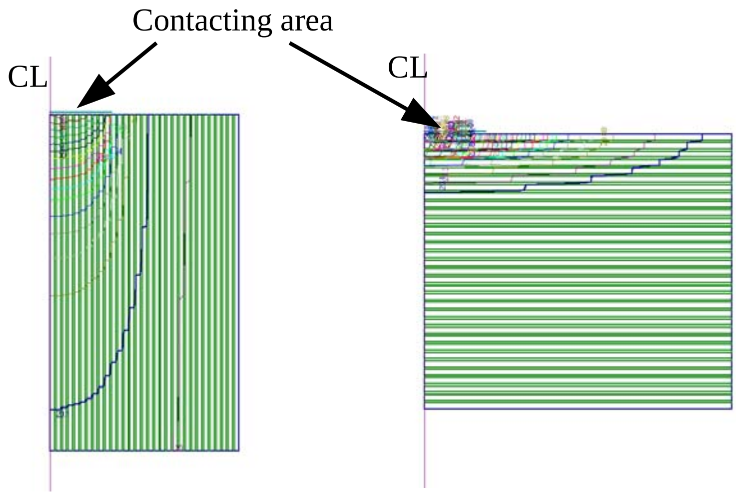

Fig. 8. Simulation of each temperature profile for the case of the probe contacting to the directions of the z-axis and the x or y-axis

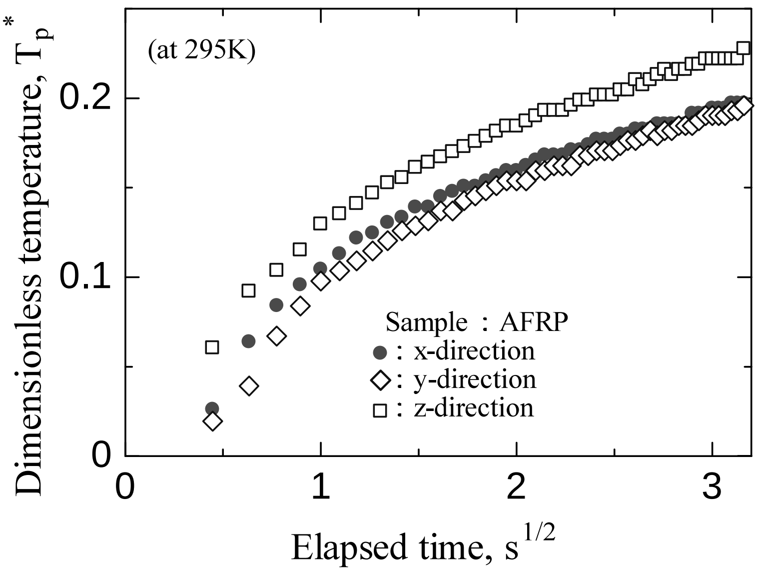

Fig. 9. Measured temperature responses for the case of the alumina-FRP

A slab of inter-metallic compound alloy, NiAl, has been produced for a sputtering substrate. The slab was 15mm-thickness, 129mm-diameter, and shown in figure 10 indicated with some measuring sites. The existence of radial distribution of thermal conductivity in the slab was revealed as shown in figure 11, while the distribution in the circumferential direction was not detected. Such valuation of thermal conductivity is caused by the size of grain boundary. The size does not only depend on the relative proportion of the two components, Ni and Al, but also result from the cooling speed when it was cast. This handy tester is useful to improve the metallurgical process of the products, and may be widely used in the industry field, because of in si-tu measurement method.

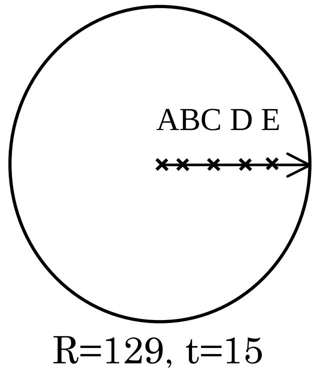

Fig.10. Testing slab of NiAl

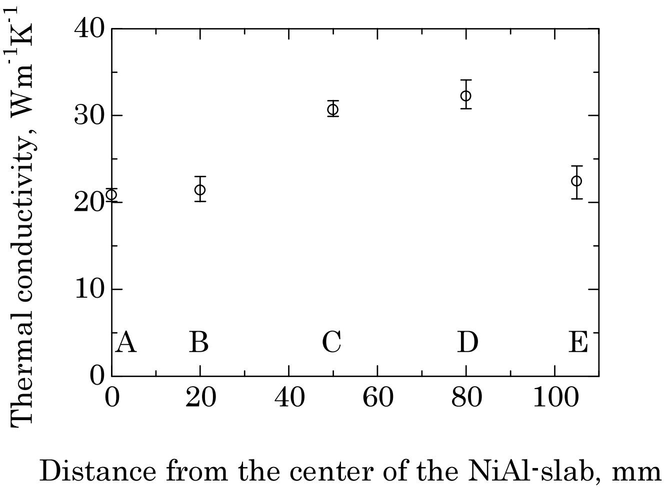

Fig.11. Radial distribution of thermal conductivity in the testing slab of NiAl

6. SUMMARY

This handy tester has been developed in order to apply the thermophysical properties measurement on the technique of quality inspection and non-destructive evaluation. The measurement of thermophysical properties by this tester is possible to make without the influence of the contact radius of the probe. The accuracy of measured thermophysical properties mainly depend on that of the probe constants determined using some standard materials. The applicable range of materials is from nonmetallic to metallic except for solids with rubber-like or porous surface. It is expected to develop the technology by this measurement method which can examine the existence of a micro-crack or the degree of the degradation of engineering solid materials.

ACKNOWLEDGMENTS

The authors express their thanks to Mr. Y. Koshikawa, student of Dept. Mechanical Systems Engineering, Yamagata University in Yonezawa, Japan, for his valuable assistance in the measurements. This research was partially supported by a Grant-in-Aid (2002) from Yamagata University Research Institute.

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